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Ionic liquid-based microwave-assisted extraction of chlorogenic acid from green coffee beans

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Microwave-assisted extraction (MAE) utilizing a hydrophilic ionic liquid, 1-butyl-3-methylimidazolium tetrafluoroborate [BMIM)][BF_4], was employed to extract chlorogenic acid from green coffee beans using Box–Behnken response surface methodology (BRSM). The yield of chlorogenic acid was considered as a function of four independent variables, namely ionic liquid concentration (M), temperature (°C), wattage (W) and time (min). By analyzing a three-dimensional surface plot of the response surface and solving the regression model equation with Design Expert software, the optimal process conditions were determined. The result shows that an extraction temperature of 90 °C, microwave power of 800 W, ionic liquid concentration ([BMIM][BF_4]) of 1 M and extraction time of 3 min were the best conditions for the extraction of chlorogenic acid. Under these conditions (IL-MAE), the maximum observed yield of chlorogenic acid was found to be 7.31%, which was higher than the conventional method of chlorogenic acid extraction (6.0%) from green coffee beans. The isomers of chlorogenic acids were found to be similar in both the conventional and ionic liquid-based microwave-assisted extracts isolated from green coffee beans. Further confirmation of the method's reliability was provided by the study, indicating that (BMIM)(BF4)-based MAE is effective for extracting chlorogenic acid from green coffee.

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Sustainability spotlight

Microwave-assisted extraction (MAE) using the hydrophilic ionic liquid 1-butyl-3-methylimidazolium tetrafluoroborate [BMIM][BF4] offers a sustainable method for chlorogenic acid extraction from green coffee beans. Experimental protocols are optimised using Box–Behnken response surface methodology (BRSM), IL-MAE improved efficiency while reducing resource use. Compared to conventional methods, IL-MAE increased yield (7.31% *vs.* 6.0%), shortened extraction time and minimized solvent waste. The use of ionic liquids eliminates volatile organic solvents, aligning with green chemistry. Microwave energy enhances efficiency, lowering environmental impact. This study highlights IL-MAE as a scalable, eco-friendly approach for bioactive compound extraction, supporting sustainability in food and pharmaceutical industries. The present works correlate with UN's Sustainable Development Goal No 12, which ensures sustainable consumption and production patterns by utilizing ionic liquids that reduce the reliance on volatile organic solvents, supporting the principles of green chemistry.

1 Introduction

Coffee, the dark, aromatic, non-alcoholic beverage, is the most widely consumed drink worldwide and holds substantial market potential. It belongs to the Rubiaceae family under the *Coffea* genus, primarily represented by two species—*Coffea arabica* (contributing 66%) and *Coffea canephora* (commonly known as Robusta, contributing 34%).[1] Both species are rich in bioactive compounds such as chlorogenic acid, caffeine, nicotinic acid, and magnesium.[2] Notably, caffeine content ranges between 1.6–2.5%, while chlorogenic acid constitutes about 7–10%. Chlorogenic acid, a class of polyphenols and an ester of quinic acid (5-*O*-caffeoylquinic acid), is known for its wide spectrum of biological activities in humans.[3] It has been reported that chlorogenic acid extracted from *Lonicera japonica* Thunb. (flowers and buds) and *Eucommia ulmoides* (leaves) plays a pivotal role in traditional Chinese medicine.[4] While solvent extraction remains a commonly used method for isolating chlorogenic acid, it suffers from limitations such as high solvent usage, extended processing time, and labor-intensive procedures.[4] In response to these drawbacks, alternative techniques such as ultrasound-assisted extraction (UAE) and microwave-assisted extraction (MAE) have been adopted by researchers.[5–7] These methods have demonstrated enhanced efficiency in both yield and quality of bioactive

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compounds. In MAE, microwave energy is converted into kinetic energy, facilitating selective heating of target compounds. This results in cellular disruption and the release of intracellular contents into the liquid phase. During irradiation, the microwave energy absorbed by the solvent significantly increases the molecular kinetic energy, thereby reducing both solvent consumption and extraction time compared to conventional methods.[8] Numerous studies have successfully applied MAE for the isolation of chlorogenic acid from *Lonicera japonica* Thunb. flower buds.[9] Despite their advantages in reducing time and improving efficiency, both UAE and MAE techniques fall short of meeting green chemistry principles, mainly due to their reliance on volatile or hazardous solvents.[9–11] To address this concern, aqueous solutions of choline-based ionic liquids (ILs) have emerged as environmentally friendly alternatives for extracting bioactive compounds from coffee and other sources.[12–20] These ILs, particularly [BMIM][BF_4], offer desirable properties such as low volatility, high thermal stability and the ability to exist as liquid salts at room temperature, making them suitable substitutes for harmful organic solvents.[21–23] Compared to conventional MAE and heat reflux extraction (HRE), the proposed IL-MAE (ionic liquid-based microwave-assisted extraction) approach demonstrates superior extraction efficiency and shorter processing times under optimized conditions.[23] UAE, for instance, relies on acoustic cavitation, which may be insufficient in disrupting tougher plant matrices such as coffee beans and often requires longer extraction durations to achieve satisfactory yields. In contrast, MAE enables rapid heating and cell rupture, but traditionally depends on organic solvents that may not align with green chemistry principles. To overcome these issues, ionic liquid-based microwave-assisted extraction (IL-MAE) has emerged as a superior alternative by combining the rapid heating advantage of microwaves with the green solvent properties of ionic liquids. Ionic liquids are non-volatile, thermally stable, and highly polar, which enhance solvent–matrix interactions and improve mass transfer. This dual benefit allows IL-MAE to not only reduce extraction time but also achieve higher yields while minimizing environmental impact. These attributes make IL-MAE particularly suitable for extracting heat-sensitive and polar compounds such as chlorogenic acid from complex matrices such as Robusta coffee beans. Moreover, evidence shows that the yield of chlorogenic acid obtained through IL-MAE, particularly from honeysuckle, is significantly higher than that of traditional methods.[23] Further enhancement in extraction yield was achieved by optimizing process parameters using response surface methodology (RSM), thereby minimizing the number of experimental trials.[24,25] Coffee is the second most traded commodity globally, valued for its rich content of functional compounds such as caffeine and chlorogenic acid. Among the two major varieties, Robusta coffee is particularly noted for its higher concentrations of these bioactives compared to Arabica. This study focuses on the application of IL-MAE using [BMIM][BF_4] to extract caffeine and chlorogenic acid from Robusta coffee beans. The goal is to optimize extraction conditions by varying parameters such as ionic liquid concentration, microwave power (wattage), extraction time, and temperature. RSM has been widely used in optimizing food processing conditions, including roasting of coffee beans,[26] pistachio nuts,[27] and hazelnuts.[28] As a robust combination of statistical and mathematical modeling, RSM facilitates the exploration of relationships between process variables and output responses.[29,30] In this study, RSM was employed to optimize the IL-MAE parameters—ionic liquid concentration, wattage, temperature, and extraction time—to maximize the yield of chlorogenic acid.

2 Materials and methods

2.1 Chemicals and reagents

A reference standard of chlorogenic acid was obtained from Sigma–Aldrich Chemical Co. (St. Louis, MO, USA). The ionic liquid, 1-butyl-3-methylimidazolium tetrafluoroborate ([BMIM][BF_4]), was purchased from Merck KGaA (Germany; product code: 4.90049.0025). Analytical-grade solvents including petroleum ether, methanol and other chemicals were procured from Qualigens Fine Chemicals (Mumbai, India) and used without further purification. Robusta cherry green coffee beans were sourced from a local market in Mysore, Karnataka, India.

2.2 Experimental design

The experiment was designed to optimize the parameters of ionic liquid-based microwave-assisted extraction (IL-MAE) for chlorogenic acid and radical scavenging activity (RSA), using water as the solvent for extraction from defatted Robusta cherry coffee. Each trial involved 1 g of coffee powder in 6 mL of distilled water, conducted at varying time intervals (1, 3, and 5 min), temperatures (50 °C, 70 °C, and 90 °C), microwave power settings (200 W, 500 W, and 800 W) and ionic liquid concentrations (0.5 M, 1 M, and 1.5 M).

The response surface methodology (RSM) optimization involved the following steps:

(1) Designing a set of statistically reliable experiments to measure the response of interest.

(2) Developing a second-order polynomial model for the response surface with optimal fitting.

(3) Determining the optimal combination of process parameters to maximize or minimize the response.

(4) Visualizing the main and interaction effects of variables through two- and three-dimensional plots.

2.3 Statistical analysis

The optimization was performed using the Box–Behnken Design (BBD) based on the principles of RSM. Statistical analysis was conducted using Design-Expert software (Version 9.0, Stat-Ease Inc., Silicon Valley, CA, USA). The BBD includes three coded levels: low (−1), medium (0) and high (+1) and is known for its efficiency and reliability in optimization studies.[31,32] Using this approach, the process parameters for chlorogenic acid extraction from coffee were optimized with the objective of maximizing bioactive compound yield. RSM is particularly suitable when the response is influenced by multiple input variables.[33–35] A total of 29 experimental runs were carried out, and the variable levels used in the BBD are summarized in Table 1.

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Table 1 Variables used in the Box–Behnken design (4-factor, 3-level design)

S.No.	Variable parameter	Symbol	Range considered		
			−1	0	+1
1	Time (min)	X_1	1	3	5
2	Wattage (W)	X_2	200	500	800
3	Temperature (°C)	X_3	50	70	90
4	Ionic liquid concentration (mole)	X_4	0.5	1	1.5

2.4 Extraction methodology

Green coffee beans were milled using a hammer mill (Comminuting Mill, Cad Mach Machinery Co. Pvt. Ltd) to achieve a particle size of 1000 ± 20 microns. The powder was defatted *via* Soxhlet extraction at 40–60 °C for 6–8 hours using petroleum ether at a 1 : 6 (w/v) ratio. The resulting defatted sample was subjected to extraction using water through 29 runs under optimized conditions in a microwave lab station (STARTS configuration with control terminal 260, Milestone, Italy) equipped with a magnetic stirrer (Magnetron: SN:133 613; 50 Hz) and an IR sensor. For each experiment, 2 g of defatted coffee was mixed with water in a 1 : 4 ratio and irradiated under the specified conditions. After extraction, the slurry was filtered through Whatman filter paper to obtain a clear extract. Details of the experimental runs with respect to variable combinations are provided in Table 2.

2.5 Conventional extraction method

For comparison, a conventional extraction was performed using 2 g of defatted coffee in 8 mL of water (1 : 4 ratio), heated at 90 °C for 3 minutes. The resulting slurry was filtered and the filtrate was collected for further analysis.

2.6 Analytical methodologies

2.6.1 Quantification of chlorogenic acid. Chlorogenic acid content was quantified using a UV-vis spectrophotometer at 325 nm (AOAC, 2000). Samples were pre-treated with lead acetate to precipitate interfering compounds. A blank was prepared under identical conditions, without lead acetate. The concentration of chlorogenic acid in the extract was determined using a standard calibration curve.

2.6.2 Determination of radical scavenging activity (% RSA). Extracts from MAE were diluted to concentrations of 20, 40, 60, 80, 100, 200, and 500 ppm. One milliliter of each sample was mixed with 4 mL of 0.1 mM methanolic DPPH solution, vortexed, and incubated at 27 °C for 20 minutes. A control (without extract) was prepared and water was used for baseline correction. Absorbance was measured at 517 nm. RSA was expressed as percentage inhibition, calculated as:

$$\%\ \text{Inhibition (RSA)} = 100 \times [\text{control OD} - \text{sample OD}]/[\text{control OD}]$$

The assay is based on the principle that antioxidants reduce the stable DPPH radical (deep violet) to the colorless compound α,α′–diphenyl-β-picrylhydrazine. The degree of discoloration indicates the radical scavenging capacity of the sample.

2.6.3 HPLC analysis of chlorogenic acid isomers. Isomers of chlorogenic acid were analyzed using HPLC, as described by Clifford[3] with slight modifications. Green coffee extracts (25 mg) were dissolved in 10 mL of 70% methanol, filtered through a 0.45 μm membrane, and injected into the HPLC system. A standard chlorogenic acid solution was prepared by dissolving 10 mg in 4 mL of distilled water and diluting it 20-fold. The HPLC system (Waters 2998, Empower software) was equipped with a C18 column (5 μm particle size, i.d. – 4.6 mm, 250 mm length). The mobile phase consisted of 0.5% trifluoroacetic acid in water and 45% acetonitrile, run on a gradient. Detection was carried out at 324 nm using a photodiode array (PDA) detector, with a 20 μL injection volume and 1 mL min^{-1} flow rate.

Table 2 ANOVA for chlorogenic acid yield

Source	Sum of squares	d*f*	Mean square	*F* value	*p*-value[a]
Model	15.46	14	1.12	2.76	0.0036
X_1	0.43	1	0.43	1.07	0.3184
X_2	0.17	1	0.17	0.41	0.5326
X_3	0.79	1	0.79	1.95	0.1840
X_4	0.011	1	0.011	0.028	0.8691
X_1X_2	0.046	1	0.046	0.11	0.7404
X_1X_3	2.86	1	2.86	7.06	0.0188
X_1X_4	0.37	1	0.37	0.90	0.3577
X_2X_3	2.31	1	2.31	5.71	0.0315
X_2X_4	0.063	1	0.063	0.15	0.7002
X_3X_4	0.50	1	0.50	1.25	0.2832
X_1^2	0.24	1	0.24	0.59	0.4537
X_2^2	0.12	1	0.12	10.31	0.5873
X_3^2	6.04	1	6.04	14.93	0.0017
X_4^2	1.78	1	1.78	4.40	0.0546

[a] Lack of fit is found to be non-significant (0.0596).

3 Results and discussion

3.1 Mathematical modeling

An analysis of variance (ANOVA) for the response surface quadratic model predicting chlorogenic acid yield from green coffee beans is presented in Table 2. The regression coefficients of the quadratic terms (X_1^2, X_2^2, X_3^2, X_4^2) were estimated using partial F-tests for individual terms, along with residual analysis. Model terms such as AC, BC and C^2 were found to be statistically significant, as indicated by "*p*" > "*F*" < 0.05, suggesting their strong influence on the response variable. The model's coefficient of determination (R^2) was approximately 0.8 (p > 0.005), indicating a reasonably good fit between predicted and experimental values. Furthermore, the signal-to-noise ratio was evaluated using Adequate Precision, where a value greater than 4 is considered desirable. In this study, the value exceeded that threshold, signifying adequate model discrimination. Model significance was also supported by higher *F*-values and lower *p*-values, emphasizing the relative importance of these variables in affecting chlorogenic acid yield.[36]

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Table 3 BBD with the observed and predicted responses for chlorogenic acid yield (%)

	Coded variable levels				Observed (Y_1)	Predicted (Y_2)
Run	X_1	X_2	X_3	X_4		
1	−1	−1	0	0	6.51	6.58
2	1	−1	0	0	6.76	6.74
3	−1	1	0	0	6.31	6.60
4	1	1	0	0	6.99	7.19
5	0	0	−1	−1	4.82	5.37
6	0	0	1	−1	4.17	5.17
7	0	0	−1	1	5.32	4.59
8	0	0	1	1	6.09	5.82
9	−1	0	0	−1	6.8	6.54
10	1	0	0	−1	6.12	6.31
11	1	0	−1	−1	6.12	6.73
12	1	0	0	1	6.65	6.86
13	0	−1	−1	0	5.51	6.01
14	0	1	−1	0	4.32	4.72
15	0	−1	1	0	5.46	4.68
16	0	1	1	0	7.31	6.76
17	−1	0	−1	0	6.54	6.35
18	1	0	−1	0	5.6	5.04
19	−1	0	1	0	4.86	5.17
20	1	0	1	0	7.3	7.24
21	0	−1	0	−1	6.79	6.10
22	0	1	0	−1	6.9	6.09
23	0	−1	0	1	5.22	5.79
24	0	1	0	1	5.83	6.27
25	0	0	0	0	6.17	6.73
26	0	0	0	0	6.87	6.73
27	0	0	0	0	6.87	6.73
28	0	0	0	0	6.87	6.73
29	0	0	0	0	6.87	6.73

The final coded regression equation (eqn (1)) representing the fitted model is given below:

$$Y = +6.73 + 0.19 \times A + 0.12{*}B + 0.26{*}\ C - 0.031{*}D + 0.11{*}\ AB + 0.84{*}\ AC + 0.30{*}\ AD + 0.76{*}\ BC + 0.13{*}\ BD + 0.35{*}\ CD + 0.19{*}\ A^2 - 0.14{*}B^2 - 0.97{*}C^2 - 0.52{*}D^2 \quad (1)$$

where Y = yield of chlorogenic acid (%), A = ionic liquid concentration (M), B = temperature (°C), C = microwave power (W) and D = extraction time (min).

It is important to note that this equation should not be used to infer the relative magnitude of each variable's influence, as the R^2 values were scaled to include only the intercept and factor units, which were not centered in the design matrix. The comparison between predicted and actual experimental values (shown in Table 3) confirms the adequacy of the proposed model, demonstrating its reliability in representing the experimental data.

3.2 Analysis of 3D response surface plots

The influence of key process parameters on the extraction efficiency of chlorogenic acid was further examined using 3D surface and contour plots generated by Design-Expert software. These plots, based on the interactions defined in eqn (1), are presented in Fig. 1.

In each plot, two independent variables were plotted along the X and Y axes, while the corresponding chlorogenic acid yield was represented on the Z axis. The remaining two parameters were held constant at their central values, as determined by the software's default setting, to isolate the effect of the interacting variables. The 3D plots clearly illustrated the combined effect of parameter interactions—particularly between ionic liquid concentration, microwave wattage, temperature and extraction time—on chlorogenic acid yield. From the surface analysis, the optimal conditions for maximum extraction were identified as:

- Ionic liquid concentration: 1 M.
- Microwave power: 800 W.
- Temperature: 90 °C.
- Extraction time: 3 minutes.

These optimized parameters were found to significantly enhance the release of chlorogenic acid from defatted green coffee beans, demonstrating the efficiency of IL-MAE under RSM-optimized conditions.

From the generated 3D response surface and contour plots, it was observed that chlorogenic acid yield increased with increasing microwave wattage and ionic liquid concentration, reaching a maximum at 1 M ionic liquid concentration and 800 W power. Among the interaction terms, the temperature–wattage relationship also exhibited a significant effect on the extraction efficiency. Chlorogenic acid content was found to be optimal at 90 °C, indicating that thermal energy synergizes with microwave energy to enhance cell wall disruption and solute diffusion. Despite p-values exceeding 0.005 for certain quadratic and interaction terms (X_3^2, $X_1 \cdot X_3$, $X_2 \cdot X_3$), the elliptical contours evident in the 2D plots—particularly around 90 °C—confirmed meaningful interactions among independent variables.[37,38] This supports the statistical robustness of the model under Response Surface Methodology (RSM).

Comparative extraction trials using ethanol, methanol and water (under MAE at 50 °C and 800 W for 5 minutes) revealed that water yielded the highest chlorogenic acid content.[39] This was attributed to its higher polarity and dielectric constant, which improved microwave absorption. The solvent performance depended on solubility parameters, mass transfer kinetics, and the nature of the solute–matrix interaction. Within the tested range, microwave power positively influenced chlorogenic acid release up to 800 W, though higher power levels were not tested due to equipment constraints. Prior studies[40,41] have reported similar trends, with increased power enhancing bioactive compound release until reaching a plateau or degradation point. However, Chemat *et al.*[42] noted that microwave power alone does not determine extraction efficiency—plant matrix characteristics and compound chemistry also play a vital role. The ionic liquid [BMIM][BF_4] appeared to enhance the extraction through its unique chemical properties. Its bromide component promotes hydrogen bonding, π–π interactions, and solvation, increasing extraction efficiency.[16,43,44] Moreover, a quadratic relationship was found between ionic liquid concentration and yield: efficiency increased from 0.5 M to 1 M, after which further increases

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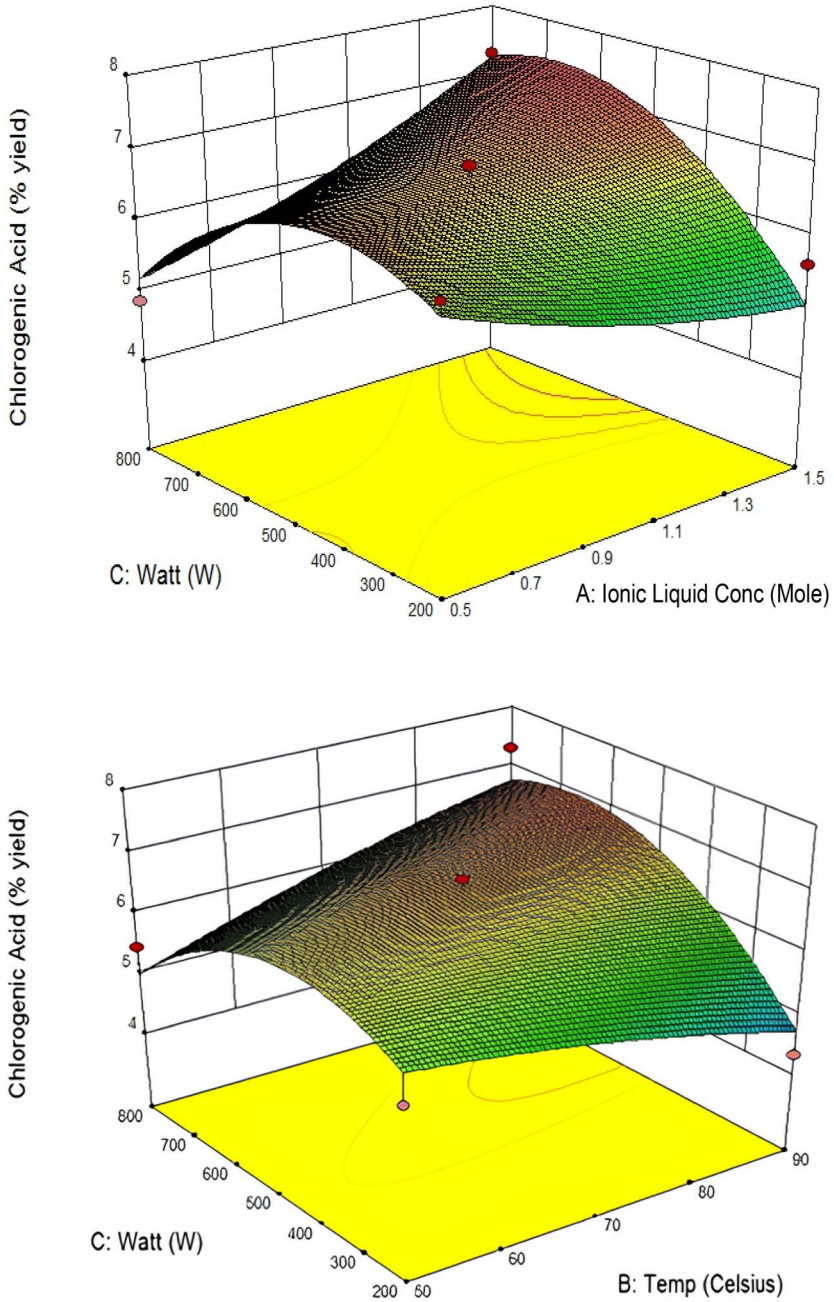

Fig. 1 Effects of (A) wattage (W) and ionic liquid concentration (mole) and (B) wattage and temperature on the chlorogenic acid yield (%) in the response surface plot.

slightly decreased extraction, likely due to viscosity-related mass transfer limitations. Maximum extraction was achieved at 3 minutes, indicating that IL-MAE significantly reduces processing time while maintaining high yield. Under optimized conditions (1 M ionic liquid, 800 W, 90 °C, 3 min), chlorogenic acid yield reached 7.31%.

3.3 HPLC analysis of chlorogenic acid isomers

Chlorogenic acid in coffee is a complex mixture of phenolic compounds, including caffeoylquinic acids (CQA), feruloylquinic acids (FQA) and dicaffeoylquinic acids (diCQA). Each class includes three isomers, defined by the position and identity of acylating residues. In this study, HPLC analysis was used to identify and quantify chlorogenic acid isomers extracted under optimized IL-MAE conditions.[45,46] The HPLC chromatogram of IL-MAE extracted fractions revealed seven distinct peaks (Fig. 2), corresponding to chlorogenic acid isomers. Notably, the chromatographic patterns were consistent with both IL-MAE and conventional extraction methods,[47,48] validating the method's selectivity. Isomer identification was performed by comparing retention times and peak areas with literature data, particularly the work of Clifford *et al.*,[47] who

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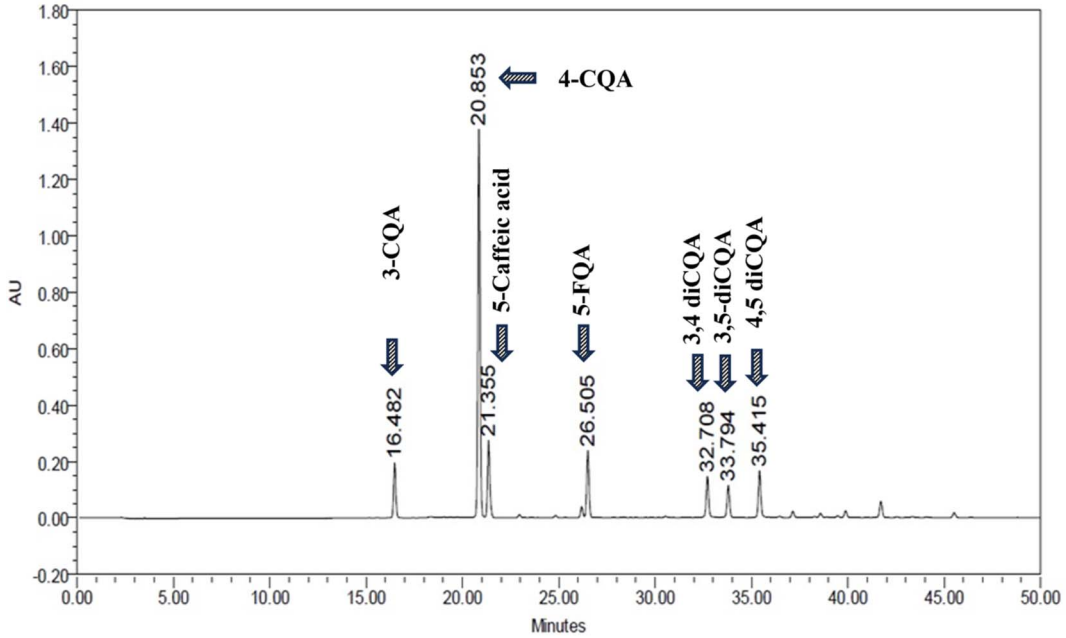

Fig. 2 Chromatogram of IL-MAE extract.

used NMR to characterize chlorogenic acid profiles. The following assignments were made:

- 1*: 3-Caffeoylquinic acid (3-CQA).
- 2*: 4-Caffeoylquinic acid (4-CQA).
- 3*: 5-Caffeic acid.
- 4*: 5-Feruloylquinic acid (5-FQA).
- 5*: 3,4-Dicaffeoylquinic acid (3,4-diCQA).
- 6*: 3,5-Dicaffeoylquinic acid (3,5-diCQA).
- 7*: 4,5-Dicaffeoylquinic acid (4,5-diCQA).

Quantitative comparison (Table 4) revealed that 3-CQA, 4-CQA and 5-FQA were dominant, comprising the majority of the chlorogenic acid profile. This aligns with previous findings in *Coffea canephora* (robusta) beans, which reported CQA isomers at 54–58%, FQA at 10–15% and lower levels of diCQA isomers.[47,48] These results confirm that IL-MAE not only enhances extraction yield but also maintains the integrity of key isomers, supporting its potential for efficient recovery of bioactive phenolics from green coffee beans.

The percentage of 3-CQA and 4-CQA was found to be around 50–60% in the IL-MAE extract compared to 30–40% in the conventional extract, but diCQA was found to be nearly the same in both. This shows that MAE was more efficient for the isolation of chlorogenic acid than the conventional process, as it reduces the solvent usage, extraction time and better extraction yield.

Among all the extracted isomers, 3-CQA and 4-CQA exhibited significantly higher concentrations across all experimental trials. This is consistent with earlier findings,[49] where increased levels of dicaffeoylquinic acid (diCQA) were linked to undesirable flavor characteristics in coffee, suggesting that a lower diCQA profile is preferable from a sensory perspective. Furthermore, it has been reported[50] that 5-CQA and other phenolic compounds are generally more abundant in immature

Table 4 Distribution of chlorogenic acid isomers (%) in total chlorogenic acids[a]

Experiments	1*	2*	3*	4*	5*	6*	7*
1	6.08	56.20	11.21	13.01	4.06	3.58	5.86
2	6.86	54.49	10.58	13.78	3.90	3.59	6.80
3	6.96	51.44	10.79	13.26	4.06	3.40	5.88
4	5.67	47.58	10.09	8.11	5.41	4.51	8.93
5	7.07	50.61	10.45	8.75	5.85	4.87	6.35
6	5.81	46.99	9.26	8.78	7.16	5.91	8.42
7	7.27	52.23	10.93	9.01	5.93	5.00	6.70
8	7.37	51.07	10.78	8.95	5.87	5.01	6.61
9	7.32	51.55	10.80	9.00	5.51	4.72	6.14
10	7.09	51.93	10.78	8.99	6.07	5.19	6.89
11	7.37	51.51	10.90	9.27	5.64	4.75	6.25
12	7.48	52.45	10.96	9.00	5.83	4.92	6.51
13	7.34	55.29	11.44	9.76	5.45	4.62	6.07
14	7.60	53.23	11.08	9.22	5.62	4.55	6.19
15	7.79	55.43	10.87	9.83	5.50	4.59	5.98
16	9.99	58.02	11.99	12.70	5.13	4.09	5.09
17	7.09	52.29	10.88	12.09	5.50	4.51	5.01
18	7.89	52.09	10.08	9.09	5.45	4.98	6.02
19	7.27	55.29	11.27	10.87	5.60	4.98	5.21
20	7.62	53.85	11.29	9.58	5.86	4.86	6.93
21	7.89	55.06	11.65	10.33	5.08	4.22	5.77
22	7.55	54.75	11.32	9.92	5.58	4.50	6.39
23	7.93	55.55	10.32	10.56	5.26	4.39	5.99
24	8.89	53.24	11.36	12.9	5.16	3.05	5.05
25	8.30	55.24	14.54	12.2	6.10	4.4	6.60
26	8.82	55.21	13.6	13.26	5.80	4.86	6.70
27	8.84	55.23	13.6	13.26	5.70	4.02	6.80
28	8.85	55.26	13.6	13.26	5.60	3.54	5.32
29	8.71	55.28	11.26	13.2	5.62	4.46	6.89
Conventional method	7.31	54.07	10.98	9.95	5.77	5.01	6.91

[a] 1*, 2*,3*,4*,5*,6* and 7* correspond to 3-caffeoylquinic acid (3-CQA), 4-caffeoylquinic acid (4-CQA), 5-caffeic acid, 5-feruloylquinic acid (5-FQA), 3,4-dicaffeoylquinic acid (3,4 diCQA), 3,5-dicaffeoylquinic acid (3,5 diCQA) and 4,5-dicaffeoylquinic acid (4,5 diCQA), respectively.

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Table 5 Comparative study of MAE and the conventional method

Exp. Set	Method of extraction	Parameters	Chlorogenic acid, yield (%)
I	Conventional refluxing method of isolation	Time (3 min); temperature (90 °C); sample: water (1 : 4)	6.0
II	IL-MAE	Time (3 min); temperature (90 °C); power (800W) sample: water (1 : 4); ionic liquid (1M)	7.31

Table 6 A comparison of chlorogenic acid extraction efficiency using various methods

S. No	Name of the extraction method	Concentration of chlorogenic acid	Reference
1	Aqueous methanol extraction	4.1%	53
2	Hot water extraction	5.07%	53
3	Boiling in water for 10 min followed by filtration	27.52 g/100 g dry extract	54
4	Boiling in a pressure cooker PS-5682 first (Austria) at 110 °C and 1.4×10^5 Pa for 10 min	36.04/100 g dry extract	54
5	Isopropanol and water (70 : 30) as solvent	4.7 g/100 g of green coffee powder	55
6	Hot water extraction	2.2 g/20 g of powder green coffee beans	56
7	Ultrasonic wave-assisted extraction	4.5 g/10 g of powder green coffee beans	57
8	Microwave-assisted extraction	2.68%/2 g of coffee bean powder	58
9	Ionic liquid-based microwave-assisted extraction	7.31%	Present system

black beans than in graded, mature coffee beans. However, defective black beans often show lower levels of 5-CQA.[51] Interestingly, the present study revealed that 5-CQA levels were consistently higher in the fractions isolated using IL-MAE compared to those obtained through the conventional method. This highlights the effectiveness of ionic liquid-based microwave-assisted extraction (IL-MAE) in preserving and enhancing the recovery of key isomers such as 5-CQA, which contribute both to antioxidant activity and health benefits.

3.4 Comparative study: IL-MAE *vs.* conventional extraction

The optimized conditions for isolating chlorogenic acid using IL-MAE were determined as 3 minutes, 800 W, 1 M [BMIM][BF_4] and 90 °C. A comparative analysis between IL-MAE and conventional water-based extraction methods is presented in Table 5. The results clearly demonstrated that IL-MAE provided a significantly higher yield of chlorogenic acid. This enhancement can be attributed to the multi-modal interactions between [BMIM][BF_4] and the chlorogenic acid molecules, including hydrogen bonding, van der Waals forces, and π–π interactions, which are known to improve extraction efficiency. The high dielectric constant and dipole moment of water in the IL-MAE system further supported microwave energy absorption, promoting rapid heating and increased solvent–matrix interaction. Moreover, microwave irradiation contributes to cell wall rupture and matrix expansion,[46–48,52] facilitating faster and more efficient release of phenolic compounds compared to conventional reflux extraction, which typically requires longer times and higher energy inputs. In support of this, Table 6 provides a comparative overview of chlorogenic acid yields obtained using various extraction techniques and solvents. The findings reaffirm that IL-MAE not only reduces extraction time but also significantly improves yield, making it a sustainable and scalable alternative for bioactive compound isolation from plant matrices.

4 Conclusion

In this study, Response Surface Methodology (RSM) was successfully employed to optimize the process parameters for the Ionic Liquid based Microwave-Assisted Extraction (IL-MAE) of chlorogenic acid from green coffee beans. The key variables optimized included ionic liquid concentration, extraction temperature, microwave power, and extraction time. The optimal conditions identified—1 M [BMIM][BF_4], 90 °C, 800 W and 3 minutes—resulted in a maximum extraction yield of 7.31%, significantly outperforming conventional extraction techniques in both efficiency and time economy. The developed quadratic model based on the Box–Behnken Design (BBD) provided robust predictive capability, as demonstrated by a satisfactory correlation coefficient ($R^2 \approx 0.8$) and significant ANOVA results. These findings affirm that the model is both valid and reliable within the studied design space. The regression equations derived through RSM effectively illustrated the influence and interactions of each factor, showing that the combined effect of parameters contributes significantly to the quadratic increase or decrease in yield. Furthermore, the IL-MAE technique proved to be rapid, reproducible, and highly effective, offering substantial advantages over the conventional reflux method. The use of a microwave-transparent, polar solvent system enhanced microwave absorption and compound recovery through efficient energy transfer and cell disruption. The ability to select suitable ionic liquids allows for targeting specific bioactive compounds across diverse food and nutraceutical matrices, enhancing extraction efficiency while reducing energy use and solvent waste. IL-MAE is well-suited for

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industrial application through integration into continuous flow and automated systems. Its modular microwave design and recyclable solvents make it both cost-effective and eco-friendly. Future work on process optimization and purification will further support its large-scale adoption. Overall, this investigation validates IL-MAE as a sustainable and scalable method for the efficient extraction of chlorogenic acid and potentially other phenolic compounds. The model's reliability, supported by experimental verification, underscores its applicability for future optimization in bioactive compound extraction.

Ethical approval

The proposed research did not entail any involvement with animal or human participants, nor was it conducted within any private or protected areas.

Author contributions

Katyaini Panday performed the experimental work, statistical analysis, and manuscript writing. Ramalakshmi Kulathooran supervised the work, contributed to manuscript writing and provided conceptualization. Subhapriya Pushparaju and Dhanapal Venkatachalam were involved in research advice, consolidation of data and manuscript writing. Divyashree Jangam Seshagiri assisted in statistical analysis & revising the manuscript. Jagan Mohan Rao Lingamallu guided the statistical analysis. All authors were involved in the article review process.

Conflicts of interest

The authors have no conflict of interest in publishing this article.

Data availability

The article includes the data that were utilized for the study.

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